

Investigation of concrete–clay interaction with regards to the thaumasite form of sulfate attack



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HIGHLIGHTS

- The aggressivity of Lower Lias Clay result in concrete deterioration exceeded that observed in standard test solutions.
- Temperature can be considered to be a secondary factor for thaumasite formation in concrete exposed to Lower Lias Clay.
- The route of TSA formation for concrete exposed to Lower Lias Clay differs from that seen in standard sulfate solutions.

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ABSTRACT

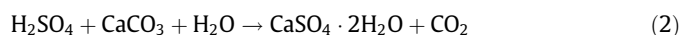
The influence of clay composition on the severity of attack due to thaumasite sulfate attack (TSA) on concrete made with CEM I and CEM I blended with 10% Limestone Filler (LF) was investigated. Concrete specimens were exposed to weathered Lower Lias Clay (LLC), for which the water soluble sulfate corresponded to the BRE sulfate class of DS-2, for 12 months at 5 and 20 °C. A parallel study was carried out in which specimens of the same concretes were placed in sulfate solutions equivalent to DS-2 and DS-4 at the same temperatures. It was found that concrete exposed to clay with sulfate design class DS-2 deteriorated at a faster rate than corresponding specimens in DS-2 solution and at a similar or greater rate to those exposed to the DS-4 sulfate solution. This suggests that the aggressivity of clay and resulting concrete deterioration could exceed what might be observed in concrete exposed to equivalent standard test solutions.

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1. Introduction

Thaumasite has been found as a product of deterioration in cement paste, mortar and concrete. Hobbs [1] quoted Erlin and Stark [2] who reported that as early as 1965 thaumasite formation was observed in sewer pipes and concrete pavements in the USA. However, in the UK the thaumasite form of sulfate attack began receiving urgent attention in March 1998 when severe damage was discovered in concrete substructures of various 30 year-old M5 motorway bridges in Gloucestershire [3]. It was also reported in buried concrete structures on the M4 in Gloucestershire, Somerset, and Wiltshire and further cases had occurred in Country Durham in NE England [4]. In addition, thaumasite has also been identified in various other locations worldwide, including Canada, South Africa, Germany, Norway and Slovenia [5,6]. In the cases of the M5 motorway, the backfill surrounding buried concrete in the dam-

aged bridges was reworked Lower Lias Clay [3]. Czerewko and Cripps [7] explain that this mudstone contains pyrite (FeS₂), a consequence of which is high levels of sulfate being present in the weathered horizons. Disturbance to the clay and its subsequent use as backfill during construction, provided access to oxygen and water which resulted in the breakdown of pyrite and a subsequent series of chemical reactions, briefly represented by reactions 1 and 2:



As shown in Eq. (1), the oxidation of pyrite results in the formation of iron hydroxide minerals, sulfate and acid. The latter in Eq. (2), reacts with calcium carbonate (CaCO₃) which is present in the clay as fossil fragments and grains, and results in the formation of gypsum (CaSO₄·2H₂O). The weathered clay therefore contains the water soluble sulfate. At the time of investigation of the bridges in 1998, the amount of pyrite had decreased by 50–75% compared with fresh clay, which resulted in an increase in the level of water

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soluble sulfate, essentially raising the clay's effective BRE sulfate design classification [8] from DS-1 and DS-2 at the time of construction to DS-3 during the service life of the bridges [3,9,10] up to the 1998 investigation, about 30 years later.

Thaumasite ($\text{CaSiO}_3 \cdot \text{CaCO}_3 \cdot \text{CaSO}_4 \cdot 15\text{H}_2\text{O}$) incorporates carbonate (CO_3), sulfate (SO_4) and silicate in the form $\text{Si}(\text{OH})_6$, associated with the calcium cation, Ca in its structure. The sulfate might be derived from sulfate associated with different cations such as magnesium, calcium or sodium, dissolved in ground water. The origin of the carbonate ions can be either from an internal source where carbonate aggregate or limestone is used, from an external source due to acid attack on carbonate or clay minerals or it can be from atmospheric carbon dioxide. In addition, the silicate becomes available due to the decomposition of calcium silicate hydrate (C–S–H) [11,12], the main binder of Portland cement.

An extensive search of the literature revealed very few studies into the interaction between buried concrete and pyrite rich clays. This is in spite of the widespread occurrence of such clays, as noted by Czerewko and Cripps [7], and the huge number of buried concrete structures. It would appear that only one field investigation has yet been carried out to assess the performance buried concrete [13]. The recommendations contained in BRE SD1 [8] and similar standards, are based upon laboratory studies involving the immersion of concrete specimens in test sulfate solutions which might be magnesium sulfate or sodium sulfate or a combination of both. This paper appears to be the first to describe TSA deterioration in buried concrete in simulated field conditions. It focuses on the preliminary results of a wider investigation into thaumasite formation and performance of concrete exposed to Lower Lias Clay.

2. Experimental work

2.1. Cementitious binders and concrete mixes

Concrete mixes were prepared using CEM I and CEM I blended with 10% limestone filler. Siliceous aggregate was used for both coarse and fine fractions. In addition, a high water to cement ratio of 0.6 was used in order to accelerate the attack process. The mix ratios of cement content to coarse and fine aggregate is 1:2.6:1.9 respectively.

2.2. Specimens preparation and exposure

In order to simulate field exposure conditions for foundations cast in shutters and then backfilled with clay, 70 mm cubes were cast and demoulded after 24 h, cured in water at 20 °C for 7 days followed by 21 days in air at room temperature. After curing, the cubes were buried in the centre of 35 by 55 by 15 cm deep containers filled with partially weathered Lower Lias Clay compacted to a density of about 1960 kg/m³; similar to the density of the backfill on the M5 motorway bridges. Each cube face was exposed to a similar volume of clay and the volume ratio of concrete to clay was 1:20. Specimens of the same concretes were also placed in sulfate solutions equivalent to Design Sulfate Class DS-2 and DS-4 listed in Table 1 and here the ratio of concrete volume to solution was 1:3. Each individual container of buried concrete or concrete in solution was placed in water tanks maintained at either 5 or 20 °C ± 0.5 °C. The solutions were renewed every 3 months for the duration of the tests. Partially weathered Lower Lias Clay was obtained from the A46 Newark to Widmerpool Highway improvement scheme from a site near Cotgrave in Nottinghamshire. Table 2 gives the main water soluble cations and anions present in the clay which were found by analysing the extracted 2:1 water–clay pore solutions according to BS 1377-3:1991 [20] using dionex analytical equipment.

Table 1
Test solution compositions.

Test solution	Solution compositions (mg/l)	
	SO_4^{2-}	Mg^{2+}
BRE DS-2	1500	500
BRE DS-4	6000	1520

The BRE water soluble design sulfate class(DS) for the clay was DS-2 as shown in Table 3, where the amount of pyrite was calculated from the difference between the total sulfur and acid soluble sulfur and it was assumed that all acid soluble sulfur is in the form of gypsum. Data about the sulfur present were found by analysing the clay according to TRL 447 [21].

2.3. pH analysis, observations and testing of specimens

The pH of the sulfate solutions and the clay were periodically determined using a Hanna HI931000 pH meter with a HI1217D electrode where, for the clay, pore solutions were extracted using 2:1 water–clay extraction according to BS1377-3:1990 [20]. The initial pH of solutions was measured and presented as the value at zero age.

The specimens were removed from the clay and sulfate solutions for visual observation and photography after 12 months of exposure for visual changes, including any color changes damage or loss of materials. Deteriorated material was sampled and analyzed using X-ray diffraction (XRD) and infra-red spectroscopy (IR) techniques in order to identify the phases present. Where deterioration products were absent, a part of the outer layer of the specimen was removed and analyzed.

Samples for XRD were air dried at room temperature to avoid any change in crystallization of deteriorated materials and then gently crushed using a porcelain mortar and pestle to pass a 150 µm sieve, in order to reduce the amount of sand particles in the powder. Material smaller than 150 µm was further reduced to 63 µm by the same method and this was then packed carefully into an aluminium holder. The XRD analysis was performed using a Philips PW 1830 X-ray generator using a copper electrode operating at 40 kV and 30 mA with a scanning speed of 2θ/min with step size 0.02° over a 2θ range of 5–60°. The database of the Joint committee for Powder Diffraction Files (JCPDF) built into WinXPow software was used to identify the different phases in the patterns.

The samples for IR spectroscopy were made as discs, using 2 mg of <63 µm ground samples mixed with 200 mg of potassium bromide which were ground together until a fine homogeneous mixture was achieved. The powdered sample was then placed in a 12 mm diameter press mould. The IR spectrum was determined using a Perkin–Elmer FT-IR 2000 spectrometer with a resolution of ±1 cm⁻¹.

3. Results and discussion

3.1. Visual observations

Figs. 1 and 2 respectively show the appearance of the CEM I and CEM I/10%LF cubes after 12 months immersion in solutions or contact with Lower Lias Clay in different conditions. In Fig. 1, deterioration of the sample is seen to have resulted in the formation of white material that is present beneath an outer skin of concrete. In the case of the 5 °C cube tests revealed that this material was easily removed to a depth of about 2 mm by scratching the surface, which suggests that attack took place under the outer layer of carbonated concrete. On the other hand, damage to the 20 °C cube was restricted to the top edge of the cube. In addition, the specimens placed in DS-4 sulfate solution at 5 °C, showed signs of deterioration at the corners, while at 20 °C white material had precipitated onto the surface. However, no signs of deterioration were recorded in cubes exposed to DS-2 sulfate solution at either temperature.

In the case of CEM I blended with 10% limestone filler in Fig. 2, the degree of deterioration was greater than that in CEM I concrete at both temperatures. It can be seen that the cubes buried in Lower Lias Clay suffered severe deterioration at both 5 and 20 °C, with spalling of the top surface and edges. This was accompanied by conversion to white mushy material at 5 °C, whereas at 20 °C the top edges and part of the sides of the cubes affected. As shown, the degree of deterioration of concrete exposed to LLC at low temperature was quite similar to, or greater than, that in DS-4 solution. However, no signs of deterioration were observed in cubes immersed in DS-2 sulfate solution at 5 and 20 °C. In addition, cubes placed in DS-4 solutions at 20 °C showed the largest deposit of white material that covered the surfaces as well as infilling cracks at the bottom corners of cubes, as shown in Fig. 2.

An overall comparison of Figs. 1 and 2 reveals that the severity of deterioration of different concrete types exposed to various

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